PROJECT NUMBER: 6505
PROJECT TITLE: Special Investigations/Methods Development
PROJECT LEADER: D. F. Ingraham
PERIOD COVERED: April, 1988

I. PROJECT ART

A. **Objective**: To investigate the role and fate of ammonia, carbon dioxide, and pH in the AB application for project ART.

B. **Results**: Examination of the data for the five tobacco types demonstrates different modes of ammonia and carbon dioxide loss in the various tobaccos. Bodied burley shows considerable volatile loss of ammonia over a 12 day period, while bodied bright shows no volatile loss and little if any loss due to reaction. Carbon dioxide loss demonstrated a spike ten minutes after sampling for all tobacco types. The change in pH proved to be tobacco family dependent with erratic behavior for the first hour after AB application.

C. **Conclusions**: The determination of AB in AB sprayed tobaccos can only be measured as a function of ammonia due to rapid loss of carbon dioxide. Rapid and highly variable changes are occurring in all tobacco types during the first hour after spraying which are not reflected in the ammonia determination.

D. **Plans**: To extend these and nicotine extraction studies to the DL blend.

E. **References**:

PM Notebook No. 8499.

II. ELEMENTAL ANALYSIS

A. **Objective**: To provide quantitative and qualitative elemental data on tobacco, cigarette paper, material evaluation samples, and special project samples.

B. **Results**:

K in ART Stem by XRF
Several samples of stem treated with monopotassium citrate were analyzed by XRF for potassium concentration. There was a discrepancy in comparing levels of potassium before and after treatment because of a dilution effect that was not accounted for. This problem has been resolved (1).

XRF Analysis at TQAF
Results from the TLA lab are reported on a dry weight basis. The monitor was recalibrated and the ash formula recalculated so that XRF results would be consistent with this (2). Support has been given to QA as needed during the initial startup period.

Thin-Film Analysis by XRF

New single element thin-film standards were used to standardize the XRF spectrometer for paper analysis. Some initial cigarette and tipping paper samples have been quantitatively analyzed for Mg, Al, Si, K, Ca, Ti, and Fe. Results look promising.

Crop Samples from 1987

XRF multielement data (Mg, Si, P, S, Cl, K, and Ca) have been acquired on bright leaf, burley leaf, and bright stem of each grade and belt. A statistical evaluation will be done to determine if there is a pattern associated with belt and grade.

C. **References**:

1. Lewis, J. Y. and S. E. Wrenn, "Correction of Analytical Data for Weight Change in Processed Materials," memo to Distribution, March 30, 1988.

2. Lewis, J. Y., "Calibration of XRF Bright Monitor on a Dry Weight Basis," memo to D. Donaher, March 22, 1988.

III. ANALYSIS OF RESIDUAL SOLVENTS IN PACKAGING MATERIALS

A. **Objective**: To provide headspace analyses for residual solvents from packaging materials and develop a QA method for the routine analysis of packaging materials.

B. **Results**:

1. Another gas chromatograph was put into service with an attached headspace sampler. This GC was equipped with a 60m DB-1 column and allowed samples to be analyzed in duplicate and comparisons made between the DB-1 and the poraplot Q columns. Standards were found to be stable over a period of a month when stored at ambient temperature in glass volumetric flasks capped with plastic stoppers.

2. Recovery studies of a standard solution added to a blank label paralleled the results obtained on the blank pack material. Recoveries were low and variable. Since these materials are likely to change with some frequency, the easiest approach would be to ignore matrix effects and obtain calibration runs in the usual manner, i.e. using a standard solution added to an empty glass vial.

3. Threshold values of the components of the volatiles solution were obtained. Standards were prepared at the threshold value, 10% above the threshold and 10% below the threshold. These were analyzed on the DB-1 column as well as on the poraplot Q column. The results have not as yet been evaluated. However it is evident that ethyl cellosolve is not observed at its threshold level on the DB-1 column but is seen on the poraplot column.

C. **Plans**: Make further comparisons of the DB-1 and poraplot columns. Complete studies of threshold studies on the DB-1 and poraplot columns.

D. **References:**

1. Picone, R., "Identification of Unknown Components in Volatile Solvents," memo to R. Dunaway, March 28, 1988.

2. Picone, R., "PTS 114," memo to Rick Dunaway, April 15, 1988.

IV. RESPONSE TO ANALYTICAL REQUESTS

A. **Objective**: To provide analytical support to R&D and Operations personnel and projects.

B. **Results**:

Analyses and investigations by the project personnel during the month of April included:

Eleven brands of Japanese cigarettes were analyzed for methoprene content. No methoprene was detected.

Three customer complaint samples were analyzed this past month. No contaminants were observed.

A capillary GC procedure for methoprene analysis is being developed to replace the packed column analysis currently being used at Universal Leaf.

Volatile compounds from two adhesive samples were identified by GC/FT-IR/MS. The major volatile component from both samples was vinyl acetate.

Several different tobaccos were analyzed for nicotine by pyrolysis and solvent extraction as part of the unextracted nicotine study. These results will be reported after more experiments are completed.

C. **References**:

1. Shelton, J. H., "Methoprene/Japanese Cigarettes," memo to D. C. Watson, April 12, 1988.

2. Shelton, J. H., "Kabat Analysis/Japanese Cigarettes," memo to T. A. Newman, April 15, 1988.